

Determining the volume expansion of the CO₂ + octane mixture using a fused silica capillary cell with in-situ Raman spectroscopy



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ABSTRACT

Accurate expansion data for CO₂ in petroleum model compounds (e.g., alkanes, cycloalkanes and aromatics) are fundamental information for CO₂ sequestration and enhanced oil recovery in oil reservoir. In this study, the volume expansion of the CO₂ + octane mixture was measured using a fused silica capillary cell with in situ Raman spectroscopy. A section of water was loaded between octane and CO₂ in the fused capillary tube to seal the octane whilst allowing the diffusion of CO₂ into the octane. Raman spectroscopy was applied during our experiments to verify that the CO₂ + octane mixture reached phase equilibrium. Moreover, a good quadratic relationship was observed between the Raman peak intensity ratio and volume expansion factor. It was verified that this method can be used not only just to obtain more accurate experimental data, but also to protract additional volume expansion factor curves or curved surfaces from measured Raman peak intensity ratio using this equation. Over an extended temperature and pressure range, this method was thus shown to be more efficient and accurate than the traditional pressure–volume–temperature methods.

1. Introduction

The injection of CO₂ has been widely accepted as an effective technique for enhanced oil recovery (EOR) in the oil industry since 1970s [1–4]. The dissolution of CO₂ in oil reduces both the viscosity and interfacial tension of oil while increasing the mobility and swelling of the oil volume in oil-bearing reservoirs, and therefore dramatically improves oil recovery [5,6]. Moreover, this technique can effectively reduce greenhouse gas by permanent storage of CO₂ in geological formations [7–9]. As the fundamental information required for CO₂ sequestration and enhanced oil recovery in oil reservoir, the volume expansion of CO₂ + crude oil and CO₂ + petroleum model compound(s) (e.g., alkanes, cycloalkanes and aromatics) mixtures have been studied over the past decades using fixed volume or variable volume pressure–volume–temperature (PVT) methods.

In the fixed volume PVT method, a certain amount of crude oil or petroleum model compound(s) and an excess amount of CO₂ are both loaded into the PVT cell successively. When the pressure in the cell remains constant at certain temperature, the mixture is considered to

have reached thermodynamic equilibrium. The volume of the CO₂ + crude oil or CO₂ + petroleum model compound(s) mixture is then measured. Welker and Dunlop [10], Jha [11], Chung et al. [12], Srivastava et al. [13], Tsau et al. [14], and Yang et al. [15] used this method to measure the volume expansion of CO₂ + crude oil mixtures. Ren and Scurto [16] determined the volume expansion of a CO₂ + decane mixture from 1.7 to 10.1 MPa at 71.1 °C. Yang et al. [17,18] investigated the volume expansion of CO₂ + alkane (hexane, octane, decane) and cyclohexane mixtures from 3 to 10 MPa at 35, 45, 50, 60 and 70 °C.

In the variable volume PVT method, certain amounts of crude oil or petroleum model compound(s) and CO₂ are loaded into the PVT cell sequentially, and the pressure is increased to transform the two-phase gas-liquid into a single phase. The cell is then continuously or intermittently depressurized to plot the pressure–volume curve of the CO₂ + crude oil or CO₂ + petroleum model compound(s) mixture. Both the saturation pressure and volume can be obtained from its transition point (the intersection point of the single-phase curve and the two-phase curve). Graue et al. [19], Li et al. [20] and Li et al. [21] used this

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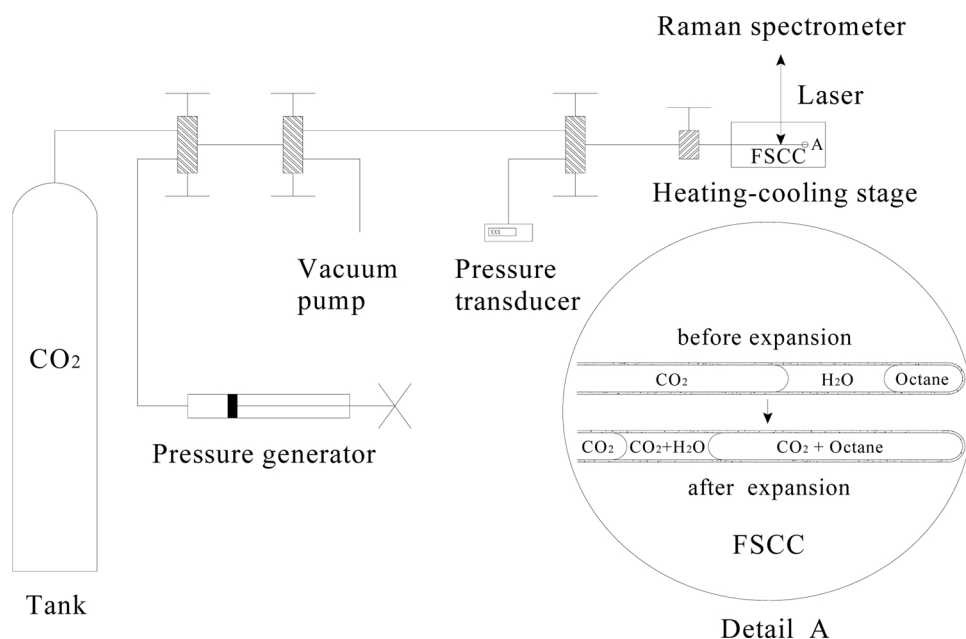


Fig. 1. Schematic diagram of in situ CO₂ + octane volume expansion measurement system.

method to determine the volume expansion of CO₂ + crude oil mixtures. Han et al. [22] measured the volume expansion of CO₂ + alkane (hexane, nonane, dodecane, tetradecane and hexadecane) mixtures with different CO₂ molar fractions.

Although a large amount of volume expansion data have been obtained for CO₂ + crude oil mixtures, the diversity and complexity of crude oil makes it difficult to obtain the patterns of volume change under different temperature and pressure conditions when crude oil comes into contact with CO₂. Previous studies have produced only a limited amount of data regarding the volume expansion of CO₂ + petroleum model compound. Moreover, all of these existing PVT methods, including both fixed volume and variable volume methods, are associated with high reagent consumption and a large temperature gradient. The fixed volume PVT method would easily cause extraction of organic materials into CO₂. The variable volume PVT method cannot easily identify the transition point for the saturation pressure–volume curve at high CO₂ molar fractions and this mixture can easily become supersaturated at lower temperatures. Therefore, improving existing experimental methods for determining volume expansion of CO₂ + petroleum model compound mixtures and obtaining accurate data is not only immediately valuable to the petroleum industry, but also important for theoretical research.

Microcell systems have been increasingly adapted to many chemical and geological applications because of their low reagent consumption, efficient heat and mass transfer and fine degree of process control. Moreover, Raman spectroscopy has been widely applied as a successful non-destructive testing technique [23–26]. The relative Raman band intensities can be used to determine substance concentrations in fluids [27,28], which is significant to further understand the changes, if any, in the composition of the tested material under investigation during experiments. In situ Raman analysis can efficiently provide this information.

In this study, a fused silica capillary cell (FSCC) [29–32], in combination with a heating-cooling stage, a pressure generator, and in situ Raman spectroscopy, was used to investigate the volume expansion of CO₂ + octane mixture under geological conditions. A section of water was loaded between the octane and the CO₂ in the fused capillary tube in order to seal the octane and allow the diffusion of CO₂ (gaseous or supercritical) into the octane [30,31]. The volume expansion of CO₂ + octane mixture was recorded with a digital camera and calculated from the accurately measured I.D. of the fused silica tube and

length of the liquid column with a micrometer at different temperatures and pressures. Raman spectroscopy was used during our experiments not only to verify that the CO₂ + octane mixture reached phase equilibrium but also to protract additional volume expansion factor plots. This study provides accurate experimental data for the volume expansion of the CO₂ + octane mixture, the fundamental and useful information needed for efficient CO₂ enhanced oil recovery.

2. Material and methods

2.1. Materials

All chemicals were purchased commercially and used as soon as they were received. Octane (C₈H₁₈, with purity of 99%) was supplied by Aladdin Chemistry Co., Ltd. (Shanghai, China) and CO₂ (with purity of 99.995%) was purchased from Pujiang special gas Co., Ltd. (Shanghai, China). Ultrapure water (resistivity 18.2 MΩ cm) was prepared using a secondary reverse osmosis system (UPT-II-20, Ulupure, China) in the laboratory. The silica capillary tubing type TSP300665 (665 μm O.D. and 300 μm I.D. with polyimide coating) was purchased from Polymicro Technologies LLC (Phoenix, AZ, USA). All valves and high pressure stainless steel tube were purchased from High Pressure Equipment Co. (Catalog No.15-11AF1, 15-15AF1, 15-9A1-30).

2.2. Volume expansion measurement system

An FSCC combined with a heating-cooling stage (Linkam, CAP500, UK), pressure generator, and confocal Raman spectrometer (Horiba JobinYvon, HR800, France) was used to investigate the volume expansion of the CO₂ + octane mixture from 30 to 80 °C and from 1 to 10 MPa (Figs. 1, S1 and S2, Supplementary material). The system temperature in the FSCC was controlled via the heating-cooling stage in conjunction with a digital temperature controller (Linkam, T95, UK, accurate to 0.1 °C), and the pressure was adjusted with a 70 MPa pressure generator and measured with a Setra 206 pressure transducer (10000 psi full scale, accurate to ± 0.13%).

2.3. Experimental procedures

The volume expansion of octane with temperature at atmospheric pressure was measured using a short FSCC (FSCC-S, ~2 cm long,

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